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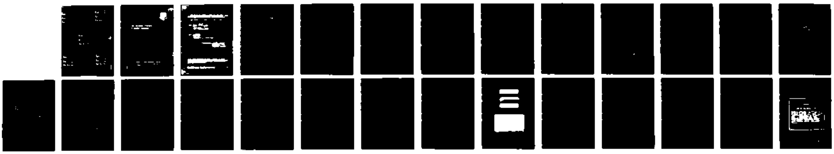

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In-House Report
April 1982

LOW PRESSURE SYNTHESIS OF INDIUM PHOSPHIDE

Joseph A. Adamski

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ROME AIR DEVELOPMENT CENTER
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Polycrystalline large grain ingots of indium phosphide have been synthesized using the direct reaction technique. Indium phosphide has been grown under various phosphorus pressures (3 to 30 atm). Several temperature profiles were used to study the effect of temperature on mobility, carrier concentrations, grain size, homogeneity, and stoichiometry. Quartz and pyrolytic boron nitride boats are used. Several experiments were performed placing the PBN and quartz boats inside boron nitride and aluminum oxide tubes in an attempt to lower the silicon contamination. Data are presented

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showing the results of different profiles, phosphorus pressures, and boat-tube materials.

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Preface

The author gratefully acknowledges the assistance of Robert C. Marshall and John K. Kennedy. He would like also to thank Joseph R. Weiner and William D. Potter for preparing the quartzware, chemicals, single crystal chips, and van der Pauw measurements.

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Illustrations

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Low Pressure Synthesis of Indium Phosphide

1. INTRODUCTION

Air Force interest in indium phosphide (InP) stems from a requirement for a lattice-matched electro-optic substrate material for 1.1 to 1.6 μm fiber optic sources and detectors. Indium phosphide is also considered a promising substrate material for optical signal processing devices such as mode-locked lasers, integrated lasers/modulators and optoelectronic switches. Single crystals grown for these substrates by the liquid encapsulated Czochralski (LEC) technique require polycrystalline starting material of the highest purity. Reduction of residual donor impurities in the polycrystalline material is essential for the growth of semi-insulating crystals or p-type materials with low carrier concentrations.

Polycrystalline large grain ingots of InP have been synthesized using the direct reaction technique under various temperatures, pressures, and boat-tube materials to determine which combination provided material with the highest purity and lowest silicon contamination. Several temperature profiles were investigated in order to determine the effect on mobility, carrier concentration, grain size, homogeneity, and stoichiometery. Experiments were conducted using quartz and pyrolytic boron nitride boats interchangeably within boron nitride and aluminum oxide tubes to reduce silicon contamination. Data are presented showing the results of different temperature profiles, phosphorus pressures, and boat-tube materials. As a result

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of these investigations, it has been shown that the highest purity indium phosphide $\mu_{77K} = 48,335\ cm^2/V\ sec$) was obtained using quartz boats in a quartz ampoule at a phosphorus temperature of 465°C and an indium temperature of 1003°C.

2. BACKGROUND

Indium phosphide is a compound composed of elements from the third and fifth columns of the periodic table. It is a direct band-gap material with an energy gap of 1.35 eV, and it has a subsidiary in the conduction band accessible to electrons at moderate electric fields. This would indicate that InP has great potential for use in transferred electron devices. Other applications are as a semiconductor for field effect transistors and as a substrate for lattice matched electro-optical devices for use with fiber optics. The two major techniques used to synthesize InP are: (1) solution growth in a horizontal Bridgman or Gradient Freeze System; and (2) direct reaction of the elements. The synthesis effort at RADC is directed to obtaining the highest purity polycrystalline material possible by the direct reaction method.

3. EXPERIMENTAL TECHNIQUE

A typical loaded quartz ampoule used in the direct reaction synthesis of InP is shown in Figure 1. The diameter is 41 mm with length of 68.5 cm. The red phosphorus is placed in the extreme right end of the ampoule. Care must be taken when loading the phosphorus to prevent any grains from adhering to the walls of the ampoule. The indium is placed in a boat of selected material and size in the left end of the ampoule. The furnace temperature profile determines the length of the ampoule, indium boat position, and melt zone.

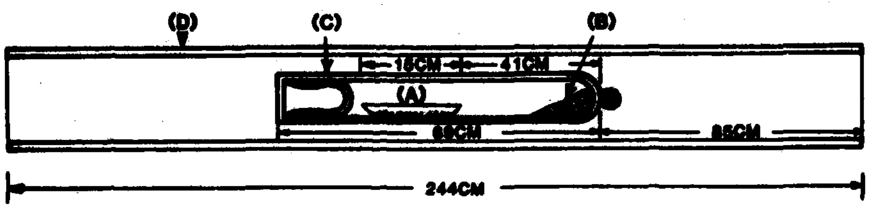

Figure 1. Synthesis of Indium Phosphide Ampoule Placements. (a) Indium; (b) phosphorus; (c) quartz ampoule (ampoule placement dependent on temperature profile); and (d) 244-cm quartz support tube

The loaded ampoule is placed in a low-temperature furnace at 300°C and evacuated to a vacuum of 1×10^{-7} Torr. A butt seal method, originally used to seal the ampoule, was suspected of introducing contamination during the high-temperature sealing process. A new method of sealing was developed which utilized a special quartz plug to fit inside the quartz ampoule. A stainless steel adapter was fabricated to couple the ampoule and vacuum system. A sketch of the vacuum system is shown in Figure 2. While under vacuum, the ampoule wall around the plug is heated until it collapses on the plug and seals. This completely eliminates any contamination from the flame or surrounding ambient. Heating during synthesis is accomplished by two Lindberg furnaces with triac type power supplies. The temperature control system utilizes Eurotherm controllers and ramp generators.

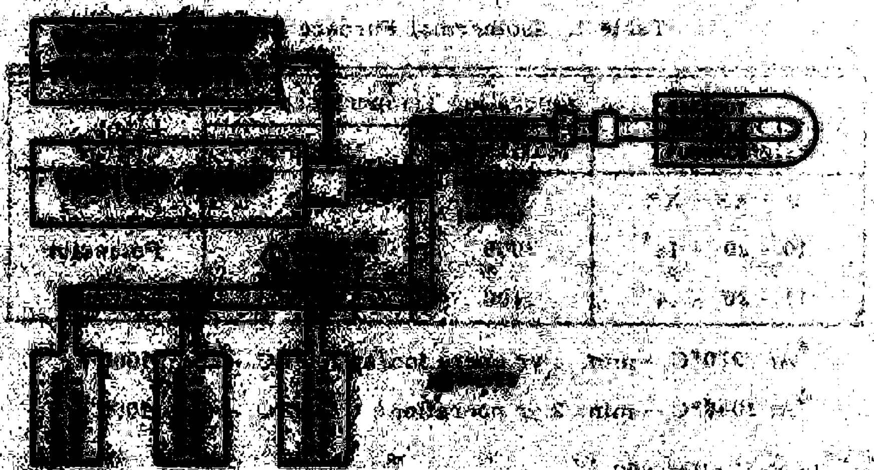

Figure 2. Vacuum-Baking System

Isothermal furnace liners (Figure 3) are used to maintain long flat zones over specific zones in the furnace system. The isothermal liners purchased from Dynatherm Corporation are heat pipes filled with either sodium or potassium for maintenance of desired temperatures over the entire pipe length. The heat pipe for the indium zone of the furnace is charged with sodium; it has an operating range of 500° to 1100°C. The phosphorus zone heat pipe is charged with potassium and it has a range of 400° to 1000°C. The heat pipes are made of Inconel 600 with very good oxidation resistance (see Table 1).

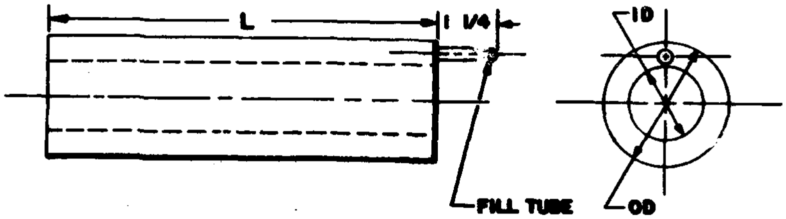

Figure 3. Isothermal Furnace Liner. The liner provides a uniform zone using a single heater. Temperature adjustment is a one-step process. Frequent profile measurements are not necessary. A flat profile is inherent to the liner. Temperature uniformity is within 1/2°C over liner length. Usable reaction zone length becomes equal to or larger than the heater length. The liner provides absolute temperature uniformity over entire length and circumference of the tube furnace wall. U.S. Patent 3,677,329

Table 1. Isothermal Furnace Liners**

Model Number Designation	Operating Temperature °C		Furnace Liner Charge
	Maximum	Minimum	
3 - XX - XX	350	200	Mercury
10 - 20 - 18*	1000	400	Potassium
11 - 20 - 24$^{\delta}$	1100	500	Sodium

*At 970°C - min. 2 yr operation/at 1000°C - min. 1000 hr

$^{\delta}$At 1040°C - min. 2 yr operation/at 1100°C - min. 1000 hr

**Inconel alloy 600
(nickel-chromium-iron)
Oxidation resistance at high temperature above 1000°C

The whole furnace system is mounted inside a hood on a motor driven table. The furnaces are moved at a specified rate in relation to the ampoule which is held stationary. An end view sketch of the furnace system (Figure 4) shows the physical relationship of the boat, ampoule, thermocouple tubes, support tube, heat pipe, furnace winding, and furnace insulation. The thermocouple tubes are placed such that a temperature profile can be taken anytime during the synthesis process; they can also be positioned to monitor the indium and/or phosphorus temperatures. The thermocouple tubes also support the ampoule and keep it from rotating during the run. When large boats are used with increased indium charges, the quartz support and thermocouple tubes are removed and the ampoule held firmly in place by a stainless steel tube attached to an eye on the end of the ampoule. A thermocouple is inserted through the tube for the purpose of monitoring the red phosphorus temperature. Another thermocouple is placed against the opposite end of the ampoule to monitor the indium temperature. The indium and phosphorus used in these experiments was 6 N's purity purchased from Metal Specialties, Inc.

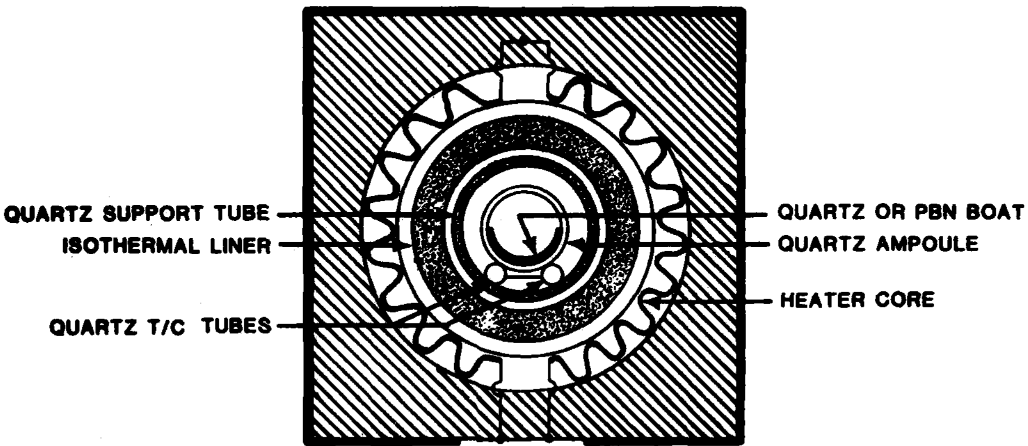

Figure 4. End View of Furnace and Components

4. EXPERIMENTATION

All quartzware are soaked and washed in deionized water with 2 percent Deconex for 1 to 2 hours, rinsed with deionized water and air-dried. The boat and plug are loaded in the ampoule, placed in a 3-zone furnace, and heated for 1 hour at 300°C. The ampoule is connected to the vacuum system and heated to 1000°C for 3 hours at 10^{-7} Torr. After cooling, the ampoule is disconnected from the vacuum and loaded at the closed end with the red phosphorus. A long-stemmed funnel is used to prevent phosphorus from sticking to the sides of the ampoule. The boat loaded with indium is placed in a specific position in the ampoule, as determined by the temperature profile. The quartz plug is placed in the ampoule and sealed under vacuum; the indium is used directly from the sealed package. A procedure for etching the indium has been discontinued because in these experiments it did not contribute to improved purity of the indium phosphide.

The over-all program to synthesize high-purity indium phosphide is comprised of four separate approaches: (1) synthesis at a high pressure of 27.5 atmospheres;[1,2,3] (2) a low indium temperature, low pressure system; (3) a low pressure high indium temperature system; and (4) a low pressure standard temperature profile using heat pipes.

A sketch of the temperature profile, ampoule and furnace arrangement for the high pressure experiments is shown in Figure 5. The indium temperature ranged from 1070° to 1150°C and the phosphorus temperature was maintained at 546°C.

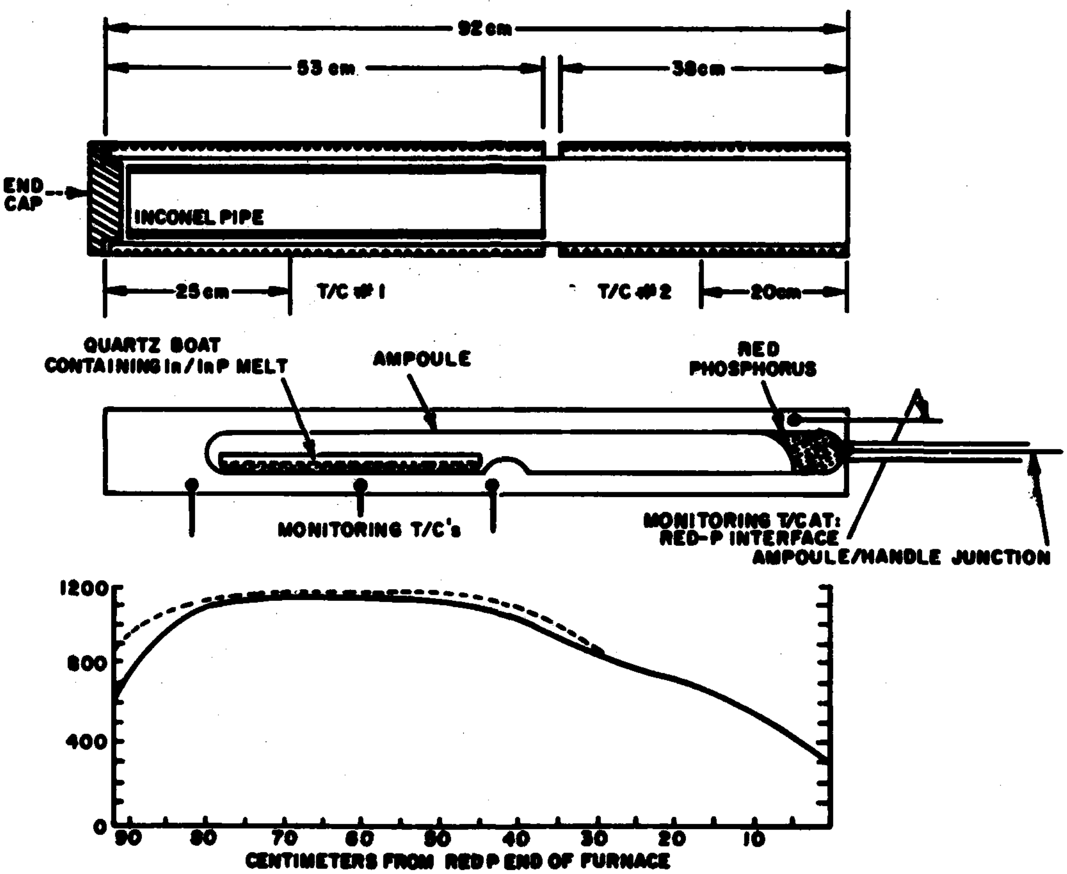

Figure 5. Indium Phosphide Synthesis at High Pressure

1. Klausutis, N., Adamski, J.A., and Sampson, J.L. (1976) Synthesis of Indium Phosphide, RADC-TR-76-305, ADA035507.
2. Fauth, T.A., and Adamski, J.A. (1979) High Pressure Synthesis of Stoichiometric Indium Phosphide, RADC-TR-79-246, ADA081875.
3. Fauth, T.A., and Adamski, J.A. (U.S. Patent No. 4,185,081).

The solid line in the temperature profile shows the initial furnace configuration. An additional winding was added to the furnace to decrease the temperature drop at each end, extending the melt zone (dotted line). Temperature fluctuations were effectively reduced with an end cap in the indium end and use of Fiberfrax in the phosphorus end. The time period for these experiments was 2 to 3 days. Typical values for mobility and carrier concentration are shown in Table 2. The possibility of explosion at these high pressures and our inability to obtain the material purity desired precluded further experimentation in favor of low pressure synthesis techniques.

The temperature profile used during the initial low pressure experiments, together with a loaded ampoule in its approximate starting position, is shown in Figure 6. The circled numbers on the abscissa indicate the various controls for the heater windings. The front of the boat containing the indium is placed at the start of the down slope. The red phosphorus zone must be at least as long as the boat because of the traveling profile. Ampoule pressure is maintained through accurate control of the phosphorus temperature.

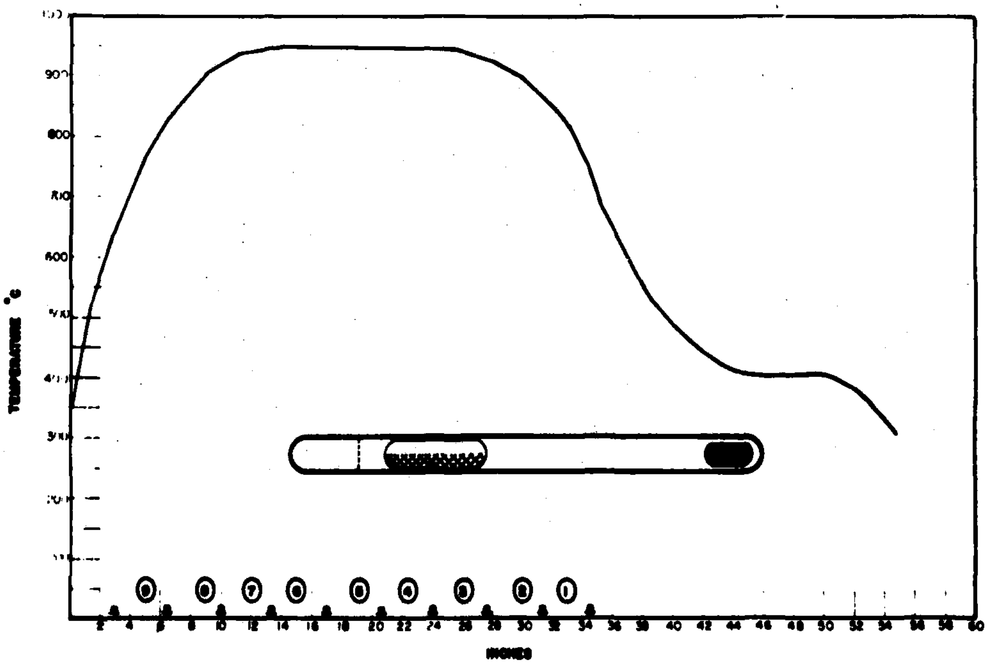

Figure 6. Indium Phosphide Furnace Profile

Table 2. Electrical Characteristics, Source of Reactants and Operating Temperatures (High Pressure)

Run No.	Boat		Ampoule		Indium		Phosphorus		Carrier Conc. (cm^3)		Mobility (cm^2/V sec)	
	Mat'l.	Source	Mat'l	Source	T°C	Source	T°C	Source	300°C	77°C	300°K	77°K
6	...	...	Quartz	GE	1062 1150	V(td)	546	V	3.9×10^{15}	3.5×10^{15}	3,880	15,610
7	...	...	Quartz	GE	1062 1150	V(td)	546	V	3.9×10^{15}	3.5×10^{15}	3,880	15,600
8	...	...	Quartz	GE	1062 1150	ICA(td)	546	V	3.9×10^{15}	3.5×10^{15}	3,880	15,610
15	Quartz	GE	Quartz	GE	1062 1150	ICA(wire)	546	V	1.2×10^{15}	8.0×10^{15}	3,270	9,690
16	Quartz	GE	Quartz	GE	1062 1150	ICA(wire)	546	P	1.0×10^{16}	6.3×10^{15}	4,830	9,420
17	Quartz	GE	Quartz	GE	1062 1150	ICA(wire)	546	P	1.3×10^{16}	9.8×10^{15}	3,360	11,520
18	Quartz	GE	Quartz	GE	1062 1150	ICA(wire)	546	P	7.4×10^{15}	5.5×10^{15}	3,270	13,500
19	Quartz	GE	Quartz	GE	1062 1150	P	546	P	2.5×10^{15}	1.6×10^{16}	3,650	13,440
21	Quartz	GE	Quartz	GE	1062 1150	MCP	546	MCP	2.8×10^{15}	2.3×10^{15}	4,295	25,810
25*	Quartz	GE	Quartz	GE	1062 1150	P	546	MCP	4.4×10^{15}	3.5×10^{15}	4,280	25,220

V - Ventron/6N tear drops
ICA - Indium Corporation of America (td)
P - Puratronic
MCP - Metal Specialties

High Pressure Furnace

*Final High Pressure Run

The indium and phosphorus temperatures were varied from 945° to 1055°C and 412° to 520°C, respectively. The traveling rate of the furnace was 12 mm per day. The quartz boats were 150 mm long and 25 mm wide. The standard ampoule charge was 150 g of 6N's indium and 45 g phosphorus. The results of several typical experiments indicating the carrier concentration and mobility at room and liquid nitrogen temperature are shown in Table 3. The highest purity material synthesized using quartz boats had a carrier concentration of 3.16×10^{15} carriers/cm^3 and a liquid nitrogen mobility of 38,912 cm^2/V-sec.

A number of indium phosphide synthesis experiments were conducted using a temperature profile (Figure 7) with a very narrow high temperature zone. The over-all temperature of the quartz ampoule and boat were kept at relatively low temperatures and a sharp temperature spike 1/2-in. wide was programmed in an attempt to increase the purity of the indium phosphide by reducing silicon contamination. The front of the boat was placed at the peak of the high temperature zone at the start of the experiments. The indium zone of the furnace was varied from 1054° to 1080°C. The phosphorus zone ranged from 430° to 469°C. With this temperature spike profile, only a small portion of the quartz ampoule and boat would be exposed to the high temperature at any period of time. Furnace travel was maintained at 1/2 in. per day. The highest purity material synthesized using this temperature profile was obtained using an indium temperature of 1060°C and a phosphorus temperature of 434°C. This material had a carrier concentration of 3.13×10^{15} carriers per cm^3 and a liquid nitrogen mobility of 31,631 cm^2 V-sec. However, since the average carrier concentration and liquid nitrogen mobility for a series of eight experiments shown in Table 4 was 6.38×10^{15} carriers per cm^3 and only 14,014 cm^2 per V-sec, respectively, it was decided to discontinue using this method.

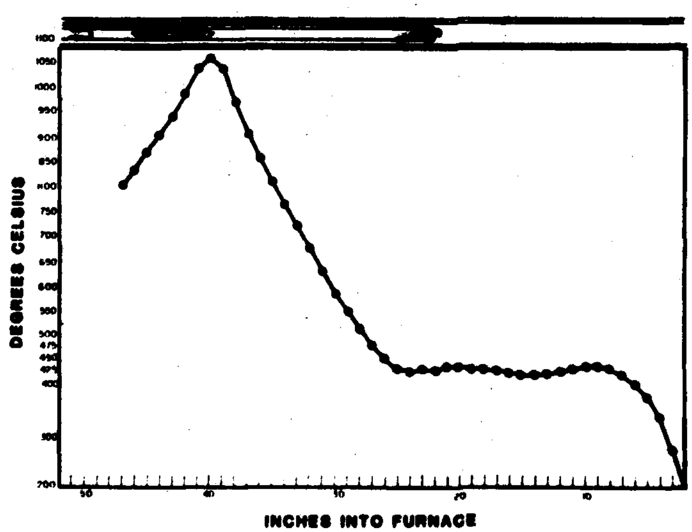

Figure 7. Synthesis of Indium Phosphide (Spike)

Table 3. Electrical Characteristics, Source of Reactants and Operating Temperatures (Low Pressure)**

LV No.	Boat		Ampoule		Indium		Phosphorus		Carrier Conc. (cm^3)		Mobility (cm^2/V sec)	
	Mat'l.	Source	Mat'l.	Source	T°C	Source	T°C	Source	300°C	77°C	300°K	77°K
1	Quartz	GE	Quartz	GE	945	P	412	P	6.15×10^{15}	4.98×10^{15}	2,820	4,509
6	Quartz	GE	Quartz	GE	1014	MCP	496	MCP	4.21×10^{16}	3.04×10^{16}	3,515	7,263
8	Quartz	GE	Quartz	GE	1017	ICA(td)	466	MCP	6.86×10^{15}	5.56×10^{15}	3,758	23,032
9	Quartz	GE	Quartz	GE	1055	MCP	520	MCP	3.16×10^{15}	2.69×10^{15}	4,287	38,912
10*	Quartz	GE	Quartz	GE	1055	MCP	520	MCP	9.06×10^{15}	7.16×10^{15}	3,061	19,914

P - Puratronic AlA

MCP - Metal Specialties

ICA - Indium Corporation of America (tear drops)

* - Exploded in Cool Down

** - Travel Rate: 1/2-in. per 24 hours

Old Furnace**

(Standard Profile)

Table 4. Electrical Characteristics, Source of Reactants and Operating Temperatures (Spike)

LV No.	Boat		Ampoule		Indium		Phosphorus		Carrier Conc. (cm^3)		Mobility (cm^2/V sec)	
	Mat'l.	Source	Mat'l.	Source	T°C	Source	T°C	Source	300°C	77°C	300°K	77°K
33	Quartz	Amersil	Quartz	Amersil	1054	MCP	430	MCP	8.94×10^{15}	6.64×10^{15}	2,453	21,276
19	Quartz	Amersil	Quartz	GE	1059	MCP	460	MCP	3.43×10^{16}	2.3×10^{16}	3,651	9,648
LL*	Quartz	Amersil	Quartz	Amersil	1060	MCP	434	MCP	3.2×10^{15}	3.13×10^{15}	4,792	31,631
16	Quartz	GE	Quartz	GE	1062	MCP	462	MCP	4.84×10^{15}	4.79×10^{15}	3,780	23,355
28	Quartz	GE	Quartz	GE	1069	MCP	460	MCP	3.75×10^{16}	1.30×10^{16}	3,711	13,353
26	Quartz	GE	Quartz	GE	1069	MCP	469	MCP	1.38×10^{15}	8.04×10^{15}	3,810	18,687
23	Quartz	Amersil	Quartz	Amersil	1070	MCP	463	MCP	1.24×10^{17}	1.07×10^{17}	2,870	3,461
17	Quartz	GE	Quartz	GE	1074	MCP	464	MCP	2.14×10^{17}	1.56×10^{17}	2,115	2,730
25	Quartz	GE	Quartz	GE	1080	MCP	447	MCP	**			

*Lincoln Lab prepared ampoule and supplied indium/ phosphorus
Travel Rate: 1 in. per 24 hours

**Heated burned out during run

Old Furnace
(Spike Profile)

A new synthesis system was designed using 2 Lindberg furnaces and 2 heat pipes. The temperature profile was programmed as shown in Figure 8. Energy to the system is provided by two triac type power supplies with associated Eurotherm controllers and ramp generators. These units, together with the heat pipes, give 2 long flat heat zones with a desired sharp temperature slope between the zones.

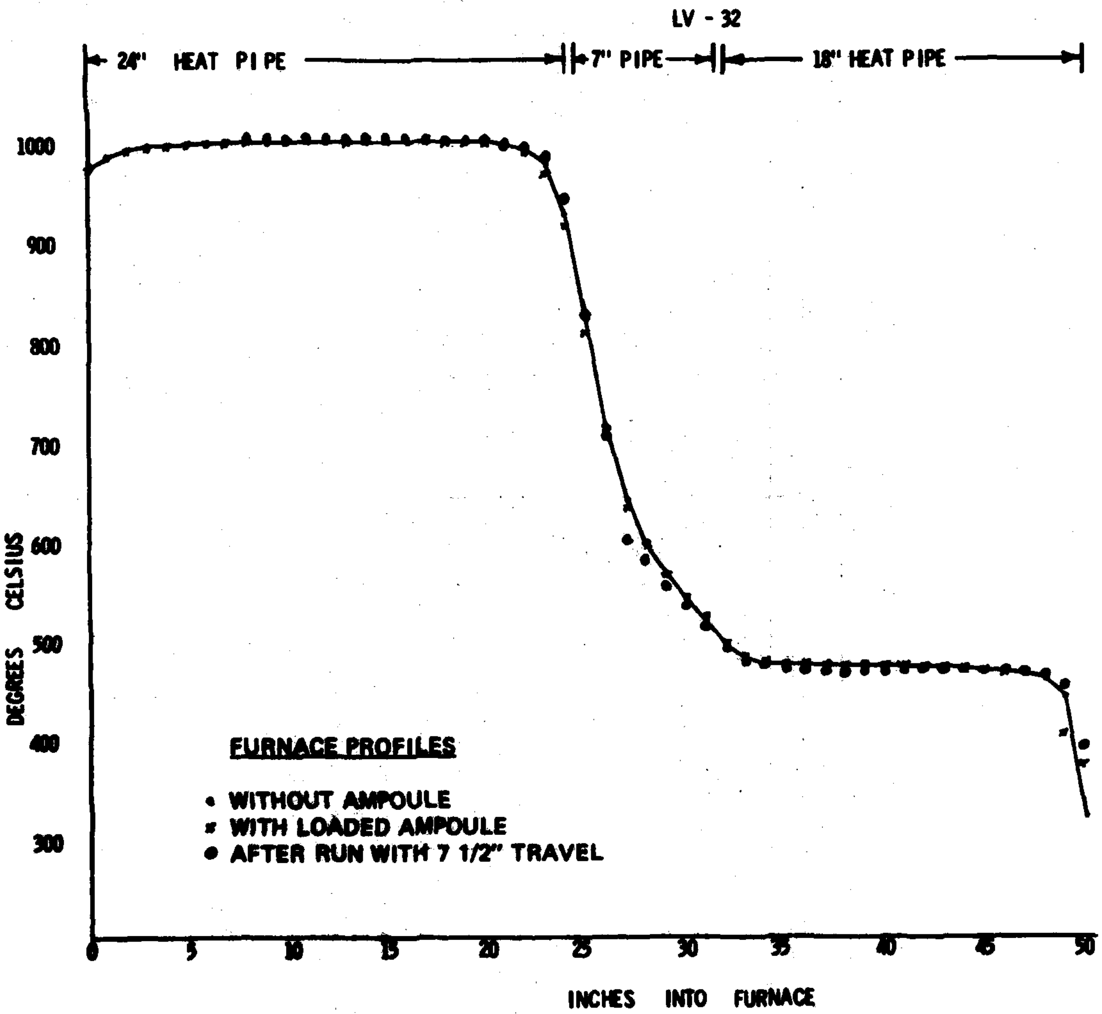

Figure 8. Furnace Profile Using Heat Pipes

During the initial start-up period, it is important always to maintain the indium at a higher temperature than the phosphorus. The indium furnace is programmed up at 100°C per hour. When a temperature of 700°C is reached, the phosphorus furnace is turned on and programmed at the same rate. This rate of increase is continued until the indium temperature reached 1003°C and the phosphorus maintained at 430° to 465°C. The furnaces are allowed to equilibrate overnight before furnace travel is initiated.

Experiments were designed around various quartz ampoule sizes and shapes and different boat materials. Boats were fabricated of quartz and pyrolytic boron nitride. In some experiments, the boats were inserted into boron nitride or alumina tubes to isolate them from the quartz ampoule. Travel rate of the furnaces for these experiments was 12 mm per day. The length of time for each experiment was 15 days. A typical polycrystalline indium phosphide ingot resulting from these experiments is shown with bottom, side, and top view in Figure 9. The right side is the first to freeze. The weight of these polycrystalline ingots ranged from 150 g to 400 g. An etched slice from one of these ingots showing typical single crystal grains used for the van der Pauw measurements is shown in Figure 10. Data showing the carrier concentration and mobility of experiments using various boat materials, shielding tubes, and phosphorus temperatures are shown in Tables 5 and 6. The highest purity material produced using these procedures was with an indium temperature of 1003°C, phosphorus temperature of 465°C using a quartz boat without a shielding tube. The resulting material had a carrier concentration of 1.8×10^{15} carriers per cm^3 and a mobility of 46,235 cm^2 per V-sec at 77°K.

5. CONCLUSIONS

Indium phosphide has been synthesized using various profiles, temperatures, pressures, and boat-tube materials to determine which combinations provided material with the highest purity and lowest silicon contamination.

The highest purity polycrystalline indium phosphide material synthesized in all the experimental techniques resulted from using a standard temperature profile (Figure 8), utilizing heat pipes, quartz boats, an indium temperature of 1003°C, and a phosphorus temperature of 465°C. The use of other boat materials and shielding tubes for elimination of silicon contamination from the quartz did not provide any measurable increase in purity as indicated from the van der Pauw measurements.

Figure 9. Typical Polycrystalline Indium Phosphide Ingot

Figure 10. Etched Slices of Polycrystalline Indium Phosphide

Table 5. Electrical Characteristics, Source of Reactants and Operating Temperatures (Heat Pipes A)*

Run No.	Boat		Ampoule		Indium		Phosphorus		Carrier Conc. (cm^3)		Mobility (cm^2/V sec)	
	Mat'l.	Source	Mat'l.	Source	T°C	Source	T°C	Source	300°C	77°C	300°K	77°K
15	Quartz	GE	Quartz	GE	1003	MCP	461	MCP	4.84×10^{15}	4.02×10^{15}	3,780	28,529
18	Quartz	Amersil	Quartz	GE	1003	MCP	464	MCP	2.2×10^{15}	1.4×10^{15}	4,196	48,235
11	Quartz	GE	Quartz	GE	1004	MCP	461	MCP	2.10×10^{15}	1.80×10^{15}	4,086	39,299
22	Quartz	Amersil	Quartz	Amersil	1004	MCP	458	MCP	1.99×10^{15}	1.68×10^{15}	3,795	39,615
20	Quartz	Amersil	Quartz	Amersil	1004	MCP	462	MCP	2.69×10^{15}	2.25×10^{15}	4,290	43,890
24	Quartz	Amersil	Quartz	Amersil	1008	MCP	458	MCP	2.71×10^{15}	2.21×10^{15}	2,934	33,782
27	PBN	UC	Quartz	Amersil	1008	MCP	456	MCP	7.0×10^{15}	4.40×10^{15}	3,742	24,915
29	PBN	UC	Quartz	Amersil	1009	MCP	458	MCP	4.81×10^{15}	3.38×10^{15}	3,595	30,013
13	Quartz	GE	Quartz	GE	1040	MCP	420	MCP	1.80×10^{16}	1.30×10^{16}	2,919	11,470
30	Quartz	Amersil	Quartz	Amersil	1057	MCP	430	MCP	1.20×10^{17}	1.30×10^{17}	2,980	3,287
12	Quartz	GE	Quartz	Amersil	1062	MCP	460	MCP	8.42×10^{15}	6.42×10^{15}	3,469	19,856
14	Quartz	GE	Quartz	Amersil	1067	MCP	462	MCP	2.82×10^{16}	2.02×10^{16}	3,311	9,217
14a	Quartz	GE	Quartz	Amersil	1069	MCP	461	MCP	8.05×10^{16}	6.16×10^{16}	3,701	5,102

MCP - Metal Specialties
PBN - Pyrolytic Boron Nitride
* - Travel Rate: 1/2-in. per 24 hours

Heat Pipe Furnace*

Table 6. Electrical Characteristics, Source of Reactants and Operating Temperatures (Heat Pipes B)

LV No.	Boat		Ampoule		Indium		Phosphorus		Carrier Conc. (cm^3)		Mobility ($cm^2/V\ sec$)	
	Mat'l.	Source	Mat'l.	Source	T°C	Source	T°C	Source	300°C	77°C	300°K	77°K
37	Quartz	GE	Quartz	GE	1002	MCP	436	MCP	2.43×10^{15}	2.45×10^{15}	3,996	39,011*
36	PBN	UC	Quartz	GE	1002	MCP	460	MCP	2.63×10^{15}	2.23×10^{15}	4,452	43,053x
38	Quartz	GE	Quartz	GE	1003	MCP	430	MCP	6.41×10^{15}	3.6×10^{15}	2,713	30,703Δ
39	PBN	UC	Quartz	Amersil	1003	MCP	430	MCP	6.52×10^{15}	4.57×10^{15}	3,029	26,874•
42	Quartz	GE	Quartz	GE	1003	MCP	434	MCP	2.60×10^{15}	2.18×10^{15}	3,579	37,258D
43	Quartz	GE	Quartz	GE N	1003	MCP N	435	MCP	4.67×10^{15}	3.86×10^{15}	3,870	27,711D
41	PBN	UC	Quartz	Amersil N	1003	MCP N	436	MCP	4.58×10^{15}	3.61×10^{15}	3,274	29,015•
44	PBN	UC	Quartz	Amersil N	1003	MCP N	436	MCP	3.05×10^{15}	2.40×10^{15}	3,471	30,616•
40	PBN	UC	Quartz	Amersil N	1003	MCP N	462	MCP	4.42×10^{15}	3.2×10^{15}	3,150	34,095
32	PBN	UC	Quartz	Amersil	1003	MCP	465	MCP	3.03×10^{15}	2.48×10^{15}	3,894	38,973
35	PBN	UC	Quartz	GE	1004	MCP	461	MCP	6.09×10^{16}	5.60×10^{16}	4,083	5,455x

* - 366 g In
x - BN tube
Δ - 400 g In
• - Alumina Tube (998)
D - Dumbbell/300 g
N - Not etched
Travel Rate: 1/2-in. per 24 hours

The use of quartz and pyrolytic boron nitride boats, interchanging them with boron nitride and alumina tubes to reduce silicon contamination, did not produce higher purity materials. It appears that an elemental contamination problem exists other than silicon. In discussion with other investigators, elements such as sulphur and/or zinc are being introduced into the growth ampoule as a contaminant from the as-received indium and possibly the phosphorus.

[illegible]

[illegible] contaminants from the indium by [illegible] flowing hydrogen.[5,6,7] Different [illegible] using both techniques. The tech- [illegible] mobility-carrier concentration [illegible]

[illegible] gradient freeze growth [illegible] system [illegible]

[illegible] LPE, [illegible]

[illegible] Crystals [illegible]

References

1. Klausutis, N., Adamski, J.A., and Sampson, J.L. (1976) Synthesis of Indium Phosphide, RADC-TR-76-305, ADA035507.
2. Fauth, T.A., and Adamski, J.A. (1979) High Pressure Synthesis of Stoichiometric Indium Phosphide, RADC-TR-79-246, ADA081875.
3. Fauth, T.A., and Adamski, J.A., U.S. Patent No. 4, 185, 081.
4. Yamamoto, A., Shinoyama, S., and Uemura, C. (1981) Silicon contamination of InP synthesized under high phosphorus pressure, J. Electrochem. Soc. 128(No. 3):585-589.
5. Kamath, G.S., and Holmes, D.E. (1980) Growth of InP by Infinite Solution LPE, Proc. of the 1980 NATO Sponsored InP Workshop, June 1980.
6. Groves, S.H., and Plonko, M.C. (1980) Liquid phase epitaxial growth of InP and InGaAsP Alloys, Proc. of the 1980 NATO Sponsored InP Workshop, June 1980.
7. Allred, W.P. (1981) High Pressure Gradient Freeze Growth of Single Crystals of Indium Phosphide, Final Report, RADC-TR-81-277, ADA107731.